THE IMPACT OF CURRENT SHAPE DURING THE CONSOLIDATION PROCESS ON PHYSICO-MECHANICAL PROPERTIES OF FUNCTIONAL Fe – Ti – C – B SYSTEM MATERIALS

Sizonenko Olga Nikolaevna, DrSc Eng., Professor, Zaichenko Andrii Dmytrovych, PhD Eng., Kovalenko Aleksandr Aleksandrovich, Pristash Nikolay Sergeevich, Torpakov Andrey Sergeevich
Institute of Pulse Processes and Technologies of NAS of Ukraine – Mykolaiv, Ukraine, E-mail: sizonenko43@rambler.ru

Abstract: *The impact of current shape during consolidation of Fe – Ti – C – B system materials on the physio-mechanical properties of obtained specimens is studied in present paper. It is experimentally found that the use of method of decrease of voltage surge on output Schottky diodes of SPS device and the decrease of operation time of overcurrent protection in power circuit of SPS generator allowed changing current harmonic composition which lead to an increase of properties of consolidated Fe – Ti – C – B specimens – porosity decreased from 15.8 to 2.8 %, hardness increased from 50 to 55 HRC, loss of mass during abrasive wear decreased from 2 to 0.5 mass. %, and bending strength increased from 750 to 1500 MPa. It is found out that increase of input power during consolidation from 1.3 to 7.5 kJ/s by changing current harmonic composition leads to an increase of physico-mechanical characteristics of Fe – Ti – C – B system metal-matrix composites. Density of consolidated specimens increased from 75 to 95 %, their hardness increased from 35 to 50 HRC, loss of mass during abrasive wear decreased from 10 to 1 mass. % and their bending strength increased from 400 to 1100 MPa.*

KEYWORDS*: SPARK PLASMA SINTERING, ELECTRIC CURRENT, CONSOLIDATION, HARDNESS, WEAR RESISTANCE, POWDER, FUNCTIONAL MATERIALS*

1. Introduction

Modern technologies, in which composite materials dispersion-hardened by ultrafine particles are used, allow significant increase of physico-mechanical properties of functional materials. Thus, analysis of literature sources shows that the use of metal matrix composite materials based on Ferrum with homogeneously distributed refractory particles of ultrafine size range allows obtainment of materials with high functional properties, prime cost of which is significantly lower than prime cost of materials based on high alloy steel [1-6].

Preservation of fine-grain structure (ultrafine size range of hardening particles) of material with density higher than 95 % is possible when consolidation is performed using methods with highly concentrated energy flows which feature higher heating rates and lower times of holding at maximal temperature if compared to conventional consolidation methods (hot pressing, sintering by ultrahigh pressure, hot isostatic pressing) [7-9]. Method of spark-plasma sintering (SPS) is one of such methods. It consists in passage of superposition of direct and pulsing currents through powder load in graphite matrix at constant mechanical load [10].

2. Problem discussion

It is known that physico-mechanical and functional properties of sintered powder materials are highly dependent on porosity, and SPS method ensures accelerated consolidation of powder materials due to quick and homogeneous distribution of spark plasma energy between powder particles during passage of electric current superposition through them [11, 12]. Therefore, studies of an impact of current shape during SPS consolidation on density and functional properties of consolidated Fe –Ti – C – B system materials is an urgent task.

The goal of present work is to study an impact of current shape during SPS consolidation of Fe –Ti – C – B system specimens on physico-mechanical and functional properties of obtained specimens.

3. Objective and research methodologies

Mixture of powders of 75 % Fe + 20 % Ti + 5% B_4C initial mass composition, which was treated by high voltage electric discharge (HVED) on kerosene with specific energy of 6.25 MJ/kg that ensured content of hardening refractory components up to 25 %, size of which lies in range between 100 and 600 nm while mean diameter of metal matrix particles was ~ 15 µm, was used as initial powder mixture for sintering [13, 14]. Phase composition (see Fig. 1) of obtained powder mixture is 75 % Fe + 5 % Ti + 5% B_4C + 14 % TiC + 6 % (Fe_3C, Fe_2Ti, TiH) according to RIR method (Rigaku Ultima IV (CuKα)) diffraction meter) [15].

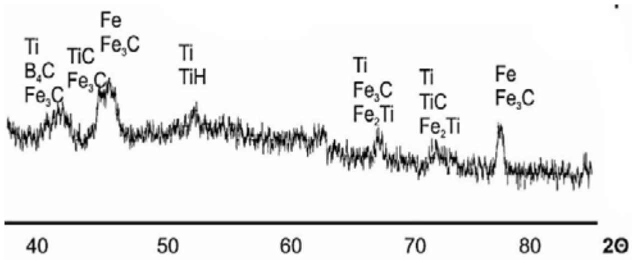


Fig. 1 *– Phase composition of Fe – Ti – C – B system powder mixture*

Consolidation of powder materials was performed in 10^{-4} MPa vacuum on “GEFEST” («ГЕФЕСТ») universal experimental complex [10], designed for usage of two electric current sources, both of which have maximum power of 10 kW. First electric current source is based on passage of superposition of direct and pulsing currents with frequency of 10 kHz at working voltage of U = 2 V, and second one is based on passage of alternating current of industrial frequency of 50 Hz at voltage of U = 10 V (method of Field Activated Pressure Assisted Synthesis (FAPAS)). Measurements of temperature, electric current intensity and voltage were performed using specially developed software. Thermal couple for measurement of temperature was introduced into matrix surface and calibrated by temperature in specimen inside matrix. Data on consolidation of selected powder mixtures on “Eran 2/1” («Эран 2/1») by direct current (conductive sintering (CS)) and “Impulse BM” («Импульс БМ») (by high voltage discharge (HVD)) was used for comparative analysis.

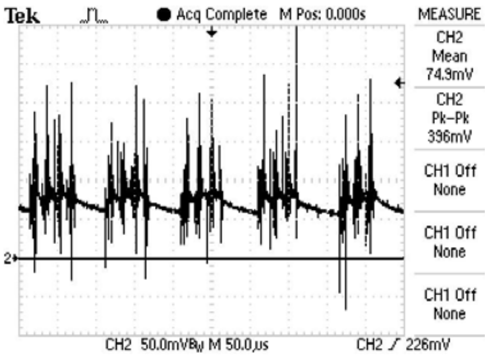
Recording of oscillograms of electric current and voltage during the use of electric current of different harmonic compositions was performed using Tektronix digital oscilloscope in order to determine energetic characteristics of consolidation process. Obtained oscillograms were processed in National Instruments Signal Express: Tektronix Edition software and imported to MathCAD software, which allowed obtainment of sintering electric power curve.

Experimental analysis of changes of consolidated materials structure was performed using computer metallography according to ISO 643:2009 using “Biolam–I” («Биолам-И») optic microscope and ImageJ software.

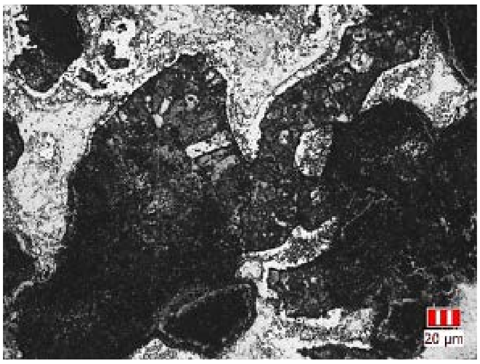
Hardness of specimens was measured according to ISO 6508-1:2013. Resistance to abrasive wear was studied on SMC-2 (СМЦ-2) friction machine using roller-block scheme. 1A1 diamond circle with AC4 80/63 grain was used as counter-body. Obtained results were compared with results of tests of HSS M2 steel.

WEB ISSN 2535-0226
PRINT ISSN 2535-0218

Oscillograms of current and voltage with corresponding microphotograph of material (see Fig. 2) were obtained during SPS consolidation of specimens with diameter of 10 mm. Amplitude of voltage was not higher than 30 V, frequency of pulses passage was not higher than 54 kHz, applied pressure was 60 MPa and time of isothermal holding was not higher than 180 s. Current amplitude in load during sintering in such regime was 1025 A (see Fig. 2, a) while DC component was 815 A and AC component was 210 A. Therefore, modulation depth of output current was 20 %. Microphotograph of consolidated Fe – Ti – C – B system specimen is shown on Fig. 2, b. This specimen has following properties: porosity – 15.8 %, hardness – 50 HRC, bending strength – 750 MPa, loss of mass during abrasive wear – 2 %.



a



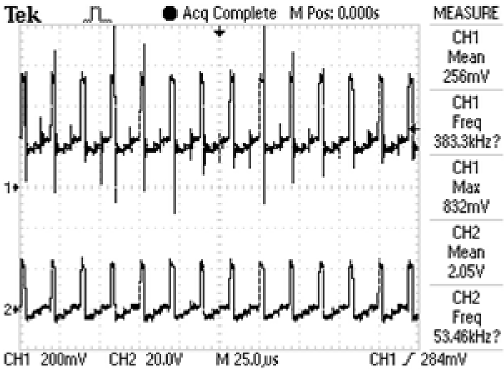
b

Fig. 2 *– Oscillogram of SPS generator output current shape (a) and microphotograph of consolidated specimen of Fe–Ti–C–B system, ×200 (white phase – Ferrum, dark phase – carbides)*

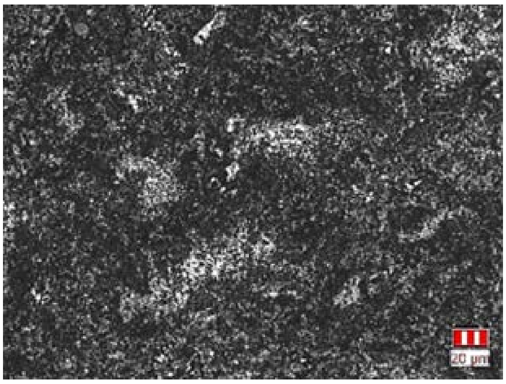
Porosity of obtained specimens was more than 15 %, which indicates insufficient power release in sintered specimen. Alter “Method for decrease of voltage surge on output Schottky diodes of device for spark plasma sintering of disperse compositions” [16] was applied and operation time of protection from overcurrent in power module of SPS generator was decreased, specimens with enhanced characteristics were obtained. Their porosity was 2.8 %, hardness – 55 HRC, bending strength – 1500 MPa, loss of mass during abrasive wear – 0,5 %. In this case, current amplitude in load during sintering was 2500 A, DC component was 1024 A, AC component was 1476 A, so modulation depth of output current was 144 % (see Fig. 3).

Thus, decrease of voltage surge on output Schottky diodes of SPS device and decrease of operation time of protection from overcurrent in power module of SPS generator leads to an increase of obtained Fe–Ti–C–B system specimen properties.

However, as it was mentioned earlier, SPS method is just one of many methods of powders consolidation by highly concentrated energy flows. Obtained data allowed comparison of SPS method after decrease of voltage urge and such consolidation methods as HVD, FAPAS and CS as well as study of peculiarities of impact of current of different harmonic composition on density and physico-mechanical properties of consolidated Fe–Ti–C–B system specimens.



a



b

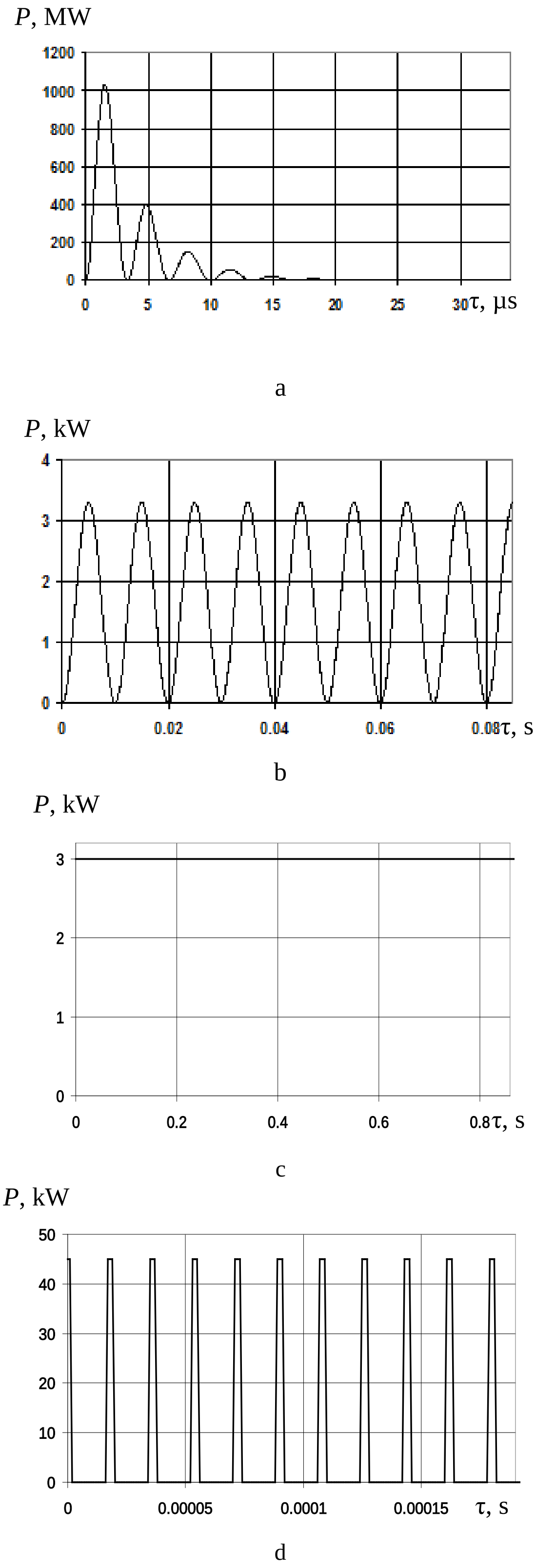
Fig. 3 *– Oscillogram of SPS generator output current shape (a) and microphotograph of consolidated specimen of Fe–Ti–C–B system after application of method of voltage surge decrease, ×200 (white phase – Ferrum, dark phase – carbides)*

Consolidation by CS, SPS and FAPAS methods was performed at temperature of 1100 °C during 180 s under the impact of 60 MPa pressure with heating rate of 10 °C/s. Compaction by HVD method was performed during 30 μs at temperature of 1100 °C and impact of pressure up to 600 MPa. As a result, dependences of porosity, hardness, loss of mass during abrasive wear and bending strength on input sintering power during 1 s, which is numerically and physically equal to electric power of sintering process for given method (see Fig. 4). For method of HVD it was 1.3 kJ/s, for FAPAS – 1.65 kJ/s, for CS –3 kJ/s and for SPS method it was 7.5 kJ/s.

It was found out that an increase of input power from 1.3 kJ/s to 7.5 kJ/s by changing electric current harmonic composition allows decreasing porosity of consolidated Fe – Ti – C – B system specimens from 25 % to 5 % due to processes of liquid metal transfer between powder mixture particles.

Increase of density of specimens, consolidated by SPS method, leads to significant increase of their hardness from ~ 35 HRC to 50 HRC, decrease of loss of mass during abrasive wear from 10 % to 1% and increase of bending strength from ~ 400 MPa to ~ 1100 MPa (see Fig. 5).

Obtained data experimentally confirm prospects of using SPS method after voltage surge decrease for obtainment of metal-matrix composites based on Ferrum, dispersion hardened by refractory components, with high values of hardness and abrasive wear resistance.



a – HVD; b – FAPAS; c – CS; d – SPS

Fig. 4 *– Values of sintering process electric power*

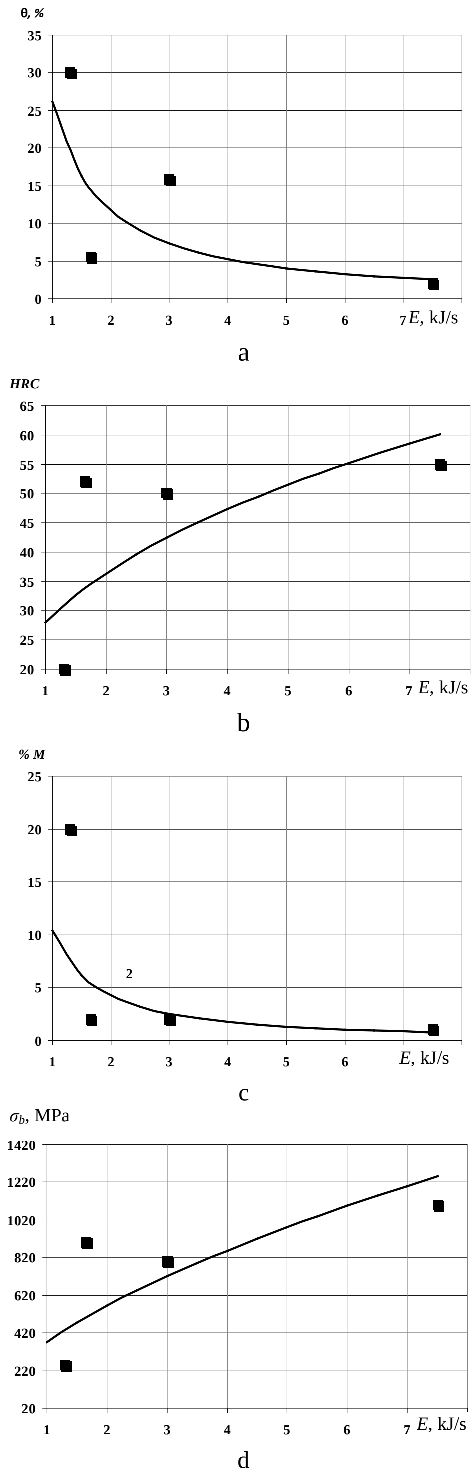


Fig. 5 *– Dependences of porosity (a), hardness (b), loss of mass (c) and bending strength (d) of consolidated materials of Fe–Ti–C–B system on sintering process input energy*

4. Conclusions

1. It is experimentally found out, that application of method of decrease of voltage surge on output Schottky diodes of SPS device and decrease of operation time of protection from overcurrent in power module for SPS generator allows changing electric current harmonic composition, namely increasing electric current amplitude in load during sintering from 1025 A to 2500 A, DC component from 815 A to 1024 A and AC component from 210 A to 1476 A, which leads to an increase of consolidated Fe–Ti–C–B system specimens properties (porosity decreases from 15.8 % to 2.8 %, hardness increases from 50 HRC to 55 HRC, loss of mass during abrasive wear decreases from 2 % to 0.5 % and bending strength increases from 750 MPa to 1500 MPa).

2. It is found out, that an increase of input electric power during consolidation from 1.3 kJ/s up to 7.5 kJ/s by changing sintering electric current harmonic composition leads to an increase of physico-mechanical properties of Fe – Ti – C – B system sintered metal matrix composites (density of consolidated specimens increases from 75 % to 95 %, hardness increases from ~ 35 HRC to ~ 50 HRC, loss of mass during abrasive wear decreases from 10 % to 1 % and bending strength increases from ~ 400 MPa to ~ 1100 MPa).

References

1. Gnesin G. Neorganicheskoye materialovedeniye: entsikl. izd. v 2 t. Kyiv, Naukova dumka, 2008, 2900 p. ISBN 978-966-00-0631-7. (Gnesing G., V. Skorokhod), (In Russian).

2. Kurganova Yu. Konstuktsionniye metallo-matrichniye kompozitsionniye materialy: uchebnoye posobiye, Moscow, N.E. Bauman MSTU, 2015, 141 p. (Kurganova Yu., A. Kolmakov), (In Russian).

3. Emamian A. Correlation between temperature distribution and in situ formed microstructure of Fe–TiC deposited on carbon steel using laser cladding. – Applied Surface Science, Vol. 258, Issue 22, 2012, P. 9025-9031. ISSN 0169-4332. (Emamian A, M. Alimardani, A. Khajepour).

4. Parashivamurthy K. Review on TiC reinforced steel composites. – Journal of Materials Science, Vol. 36, Issue 18, 2001, P. 4519–4530. ISSN 0022-2461. (Parashivamurthy K., R. K. Kumar, S. Seetharamu, M. N. Chandrasekharaiah).

5. Bagliuk G. Structural features of hot-forged carbide steel (high-speed steel–titanium carbide). – Powder Metallurgy and Metal Ceramics, Vol.48, Issue 1–2, 2009, P. 34–37. ISSN 1068-1302. (Bagliuk G., I. Martykhin, T. Pavlygo, G. Serdyuk, V. Tkach).

6. Zaichenko A. Udoskonalennya procesiv elektrorozryadnoyi obrobky shyhty ta iskro-plazmovoho spikannya dlya otrymannya vysokoznososiykyjh karbidostaley. Autoref. of PhD Thesis, speciality 05.16.06 "Powder metallurgy and composite materials", Kyiv, 24 p. (Zaichenko A.), (In Ukrainian).

7. Raichenko A. Osnovy processa spekaniya poroshkov propuskaniem elektricheskogo toka, Moscow, Metallurgiya, 1987, 129 p. (Raichenko A.), (In Russian).

8. Hong P. Spark Plasma Sintering of Si3N4-Based Ceramics. Doctoral Dissertation. Department of Inorganic Chemistry Stockholm University S-10691, Stockholm, Sweden, 2004,104 p. (Hong P.).

9. Ragulya A. Konsolidirovanniye nanostrukturniye materialy, Kuiv, Naukova dumka, 2007, 376 p. (Ragulya A., V. Skorokhod), (In Russian).

10. Patent 101575 Ukraine, IPC B22F 3/14, B22F 3/105, B30B 15/02. Device for spark-plasma sintering of powders. Sizonenko O.M., Ivliyev A.I., Raichenko O.I. et. al. No. 201200957, appl. 30.01.12, publ. 10.04.12, Bul. No. 7, 5 p.

11. Kang S.-J. L. Sintering. Densification, Grain Growth and Microstructure, Amsterdam, Elsevier, 2005, 265 p. (Kang S.-J. L.).

12. Grigoriyev Ye. Elektroimpulsnaya tekhnologiya formirovaniya materialov iz poroshkov, Moscow, MePhI, 2008, 152 p. (Grigoriyev Ye., B. Kalin), (In Russian).

13. Sizonenko O. N. Osobennosti elektrorazryadnoy obrabotki smesey na osnove poroshkov Fe i Ti razlichnogo sostava. – Visnyk ukrainshoho materialoznavchogo tovarystva. Vol. 6, 2013, P. 104 – 111. (Sizonenko O. N., N. S. Pryistash, E.I. Taftai et al.), (In Russian).

14. Sizonenko O. N. Vliyaniye vysokovoltnoy obrabotki poroshkovykh kompozitsiy sostava Fe-Ti-C i Fe-Ti-B-C na izmeneniye ikh elektricheskogo soprotivleniya. – Trudy Instituta problem materialovedeniya im/ I. N. Frantsevicha NAN Ukrainy. Seriya "Kompositsionniye, sloistiye i gradiyentniye materialy i pokrytiya". Elektricheskiye kontakty i elektrody, 2014, P. 129-133. (Sizonenko O. N., N. S. Pristash), (In Russian).

15. Chung F. Quantitative Interpretation of X-ray Diffraction Patterns. I–II. Appl. J. Crystallogr., Vol. 7, 1974, P. 519–531. (Chung F.)

16. Kovalenko A. Metod umensheniya vspleska napryazheniya na nykhodnyhk diodakh Schottky ustrojstva iskroplazmennogo spekaniya dispersikh kompozitsij. – Tekhnika ta elektrofizyka vysokykh napruh. Visnyk NTU "KhPI", 2016, No. 36 (1208), P. 31-36. ISSN 2079-0740. (Kovalenko A.A., O.N. Sizonenko), (In Russian).